NOTICE: This document contains references to Varian. Please note that Varian, Inc. is now part of Agilent Technologies. For more information, go to **www.agilent.com/chem.**

Agilent Technologies

Sample Analysis Report
Number 39

Measuring Relative % Reflectance of Small Samples in a Cary 50 Spectrophotometer

Dr. Robert Keighley
Varian Limited 10 Mead Road, Yarnton Oxford. 0X5 1QU

Introduction

The Very small beam geometry, 1.5 nm bandwidth, and very high intensity flash source of the Cary 50, coupled with a long pathlength Czerny-Turner monochromator design and ultra-fast scanning capability, make the Cary 50 a uniquely powerful instrument when coupled to fiberoptics microvolume devices such as the Hellma Traycell. The ease of use is further enhanced due to the room-light immunity of the system, which removes the need to close the sample compartment when taking a reading. The standard Cary 50 microcell holder provides sufficient precision of alignment to easily interchange between microcell and conventional cuvettes.

The Cary 50 uses a Xenon Flash source lamp, which is so durable it should never need replacement. The instrument requires no warm-up time, and the light beam, although intense during the sub-microsecond flash, is unlikely to photo-degrade sensitive samples as the average intensity is low, and the sample is only ever illuminated with monochromatic light.

The Hellma Traycell is undoubtedly useful for measuring small samples of moderately highly absorbing liquids, but it may have an additional role as a device for measuring reflectance of small samples as it can provide a relative %R value with comparison to it's own standard mirror. Alternatively a calibrated known mirror could be used in place of the standard cap mirror of the Traycell. To investigate this capability, a variety of samples were presented. These were small electro-optic devices recovered from CD drives.

Instrumentation

All measurements were made using a standard Cary 50 fitted with the high precision (Eclipse) cell holder (0210167200) which has been installed and aligned following the protocol given in the Cary 50 TrayCell installation and operation guide*

Figure 1 *The Hellma TrayCell™ provides a simple and very precise solution to low-volume measurements, and also provides a platform for small sample %R measurements*

Figure 2 The award winning and versatile Cary 50 Spectrophotometer

Conditions

The Cary 50 is designed to measure specular reflectance from a range of samples and the software includes %R as a standard measurement. The samples were scanned using the standard scan software module, set to record a baseline (100%R) using the TrayCell standard mirror as the reference. A calibrated reference mirror could be used as an alternative.

Figure 3 *Setup screen for measurement parameters.*

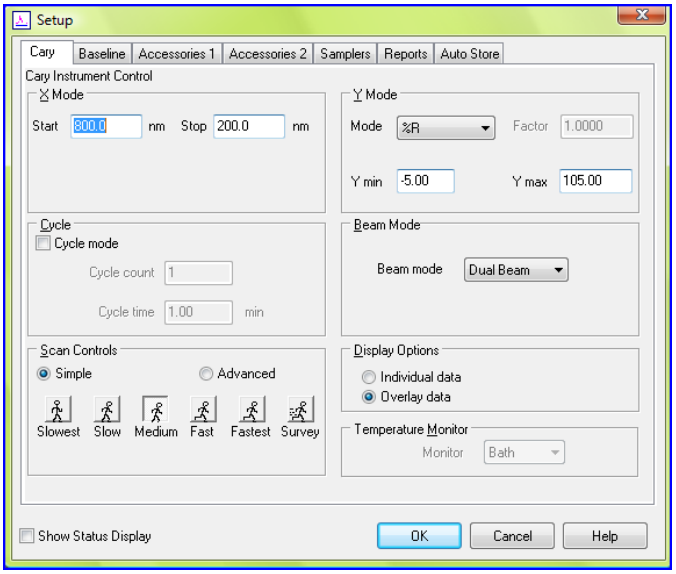
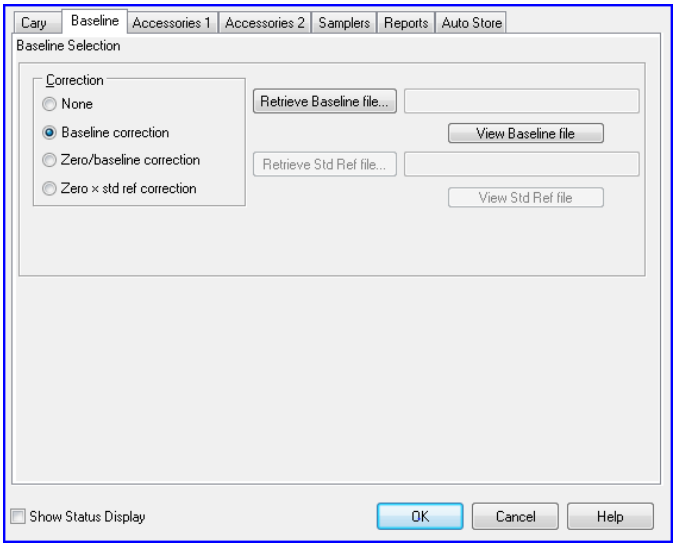


Figure 4 *Setup on Baseline screen*

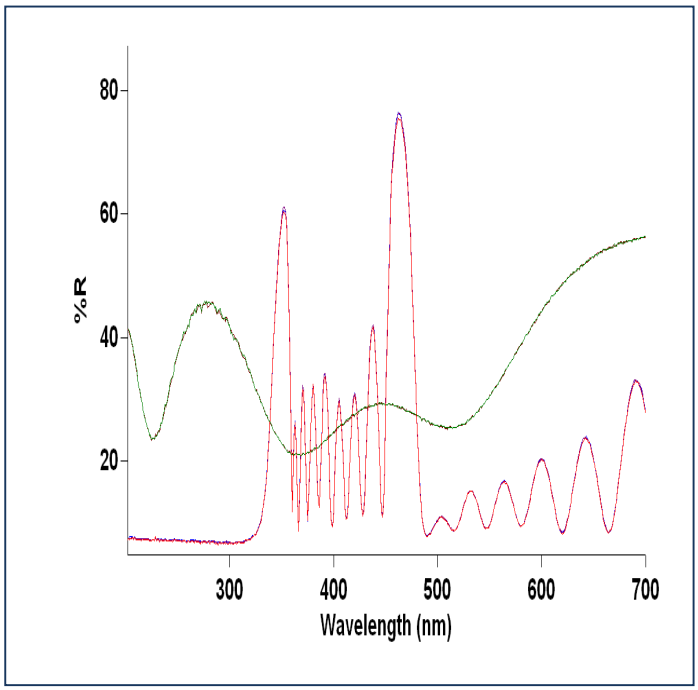


The standard mirror on the TrayCell was removed and replaced with a sample

Figure 5 *TrayCell with sample mounted for analysis.*

Results

Figure 6 *A beamsplitter (6 mm x 2 mm) from a 1990's CD drive, compared with a beamsplitter from a modern portable CD player. (in Figure 5)*



The green trace is the older beam-splitter design compared with the smaller, coated modern device in the red trace. The reproducibility of the test is seen, as these are each overlaid results of three scans.

Figure 7 *Reproducibility and precision of the results can be investigated by using the zoom tool to determine the variance between the traces repeated under identical conditions.*

Repeatability of results with sample removal and replacement between readings:(detail from Fig 6)

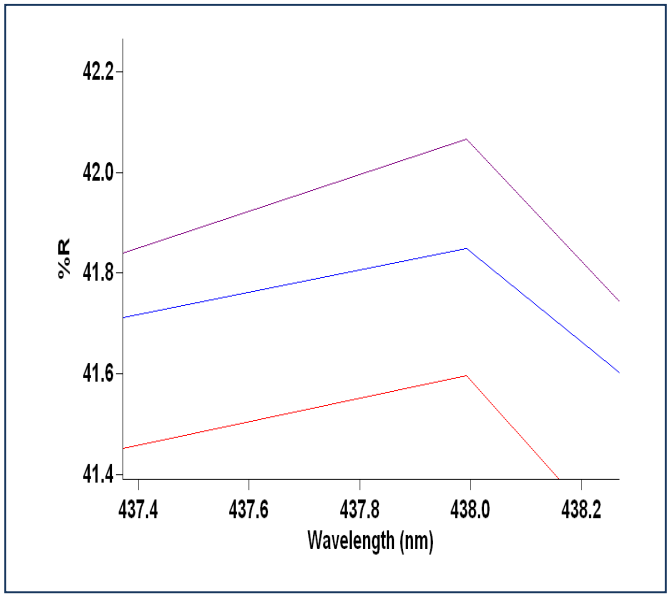
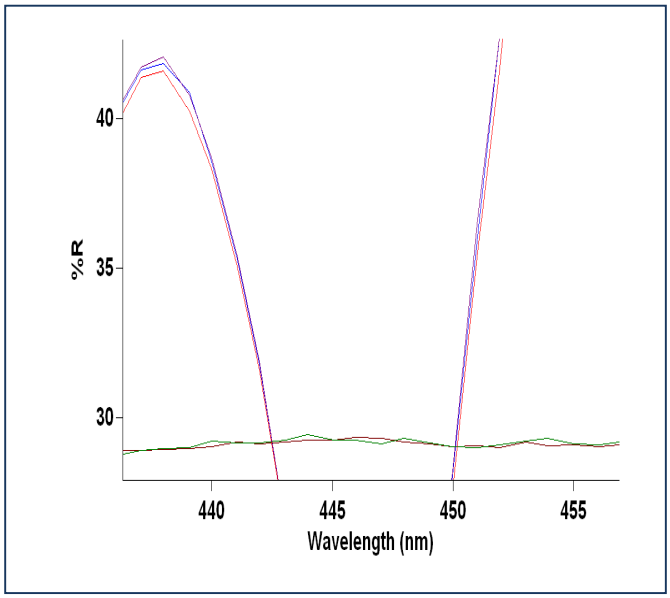


Figure 8 Full zoom to show repeatability

The Measured Variance is only about 0.4% Reflectance



on the narrow 438 nm peak of the coated beamsplitter

Discussion

The samples shown are difficult to measure using conventional reflectance accessories due to their small size. For samples like these the Cary 50 and Traycell combination gives an excellent platform to measure the samples with an acceptable precision. Samples could also be measured with the reference or standard mirror behind the sample to give a reflection/absorbance characteristic for the sample.

These data are the actual untouched experimental results obtained on Monday 18th June 2007

For further information, contact your local Varian Sales Office. 01865 291500